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WADC TECHNICAL REPORT 52-161

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DEVELOPMENT OF HIGH-STRENGTH, HEAT-RESISTANT PHENOLIC LAMINATING RESIN

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JULY 1952

WRIGHT AIR DEVELOPMENT CENTER

[redacted]

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Cincinnati Testing and Research Laboratories

July 1952

Materials Laboratory
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Wright Air Development Center
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United States Air Force
Wright-Patterson Air Force Base, Ohio

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FOREWORD

This report was prepared by the Cincinnati Testing and Research Laboratories, Cincinnati, Ohio, under U.S.A.F. Contract No. W 33(038)ac21090. The contract was initiated under the Research and Development Order No. R 614-12, Aircraft Structural Plastic Laminate, and it was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with Mr. H. S. Schwartz acting as project engineer.

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ABSTRACT

The development of a high temperature resistant glass base phenolic laminate is described. Flexural ultimate strength values of the laminate with 181-114 glass fabric at 500° F after one-half hour at 500° F pressed at 15-35 psi pressure average 40,000 psi. This is for 1/8 in. thick laminates up to 36 in. x 36 in., the larger size panels being bag molded. On these panels bag molded by North American Aviation, values of 35,000-45,000 psi were obtained at elevated temperatures. The higher values for bag molded panels have been obtained on 36 in. x 36 in. direct compression panels but they have not been obtained consistently to date. On direct compression panels 36 in. x 36 in. values from 25,000-30,000 psi have been obtained consistently at 500° F after 1/2 hr at 500° F. Values up to 55,000 psi can be obtained consistently for small laminates (such as those used in experimental compressor blades for jet engines) made at pressures in the range of 200-300 psi.

Initially these large panels 18 in. x 18 in. x 1/8 in., and 36 in. x 36 in. x 1/8 in., when handled in the same manner as 6 in. x 6 in. x 1/8 in. panels, resulted in sheets with deteriorated surfaces and lowered flexural strength at elevated temperatures, (20-25,000 psi). Since the material is applicable where larger sections with good high temperature resistance is necessary, work was continued in attempting to make satisfactory large panels.

It was found that different methods of impregnation of the resin on the glass and different methods of handling were necessary. Work along these lines has reached the point where the objective of 40,000 psi flexural strength at 500° F after 1/2 hr at 500° F has been obtained on specimens from panels 36 in. x 36 in. x 1/8 in. (Bag molded).

Fabrication techniques for parts of various sizes and configurations are described.

Some data on long-time temperature tests are reported. Some preliminary evaluations of laminates with various finishes on the glass cloth are presented.

The material used in making all panels except those for checking the finishes were made on production size equipment.

The Security Classification of the title of this report is UNCLASSIFIED.

PUBLICATION REVIEW

Manuscript Copy of this report has been reviewed and found satisfactory for publication.

FOR THE COMMANDING GENERAL:

M. E. SORTE
Colonel, USAF
Chief, Materials Laboratory
Directorate of Research

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INTRODUCTION

Early in 1947 a representative of the Cincinnati Testing and Research Laboratories supplied a small sample of a high-pressure phenolic fiber glass laminate to the Materials Laboratory, **Wright Air Development Center. Flexural tests** of this material indicated excellent properties at elevated temperatures. In view of the many specific requirements for low-pressure laminates with high temperature resistant properties, a Purchase Order 33-096-48-2981-E covering high temperature resistant glass fabric base laminate was issued in an attempt to obtain experimental quantities of laminated flat sheets of various thicknesses, made up of the best material at low-pressure similar to the high-pressure samples that could be obtained in a short time. The material was to be used for such applications as missile radomes, antenna housings, and other structural parts subject to aerodynamic heating, ducting for hot gasses, and plastic parts of, or located near, jet engines.

Because of the techniques employed in the lower pressure fabrication, production is simplified and tools are less expensive.

The original high-pressure laminates were made from a standard resin which was modified in order to obtain the characteristics necessary for low-pressure moldings. This resin laminated with 181-114 glass fabric at 100 psi pressure and having a resin content before laminating of 40 - 43% and acetone extractable of 91% gave the following flexural strength properties for flat panels approximately 6 in. x 6 in. x 1/8 in. size.

	Room Temperature	400° F	500° F
Proportional limit, psi	30,700	20,300	19,000
Yield Strength, 0.2% offset psi	55,700	48,900	46,100
Ultimate Strength, psi	55,700	50,200	46,100
Modulus of Elasticity, psi x 10^6	3.59	3.72	3.73

In view of the promising results obtained, a contract W-33-038-ac-21090 was entered into by Cincinnati Testing and Research Laboratories with the object of developing a material moldable at still lower pressures (15 psi) in large sizes and having mechanical properties at elevated temperatures equal to the above material together with satisfactory electrical properties.

The phenolic laminating resin designated CTL-91-LD has been developed with which a glass fabric laminate of good quality can be low-pressure molded at 15 psi.

Inasmuch as phenolic resins react in a condensation reaction, water is released in curing. The removal of both the residual volatiles and the volatiles of condensation is necessary in order to obtain panels with no deterioration. On small areas this is accomplished more readily because the volatiles do not have as great a distance to move. This is not the case as the panel or part increases in size. Since difficulty was experienced in making larger panels, work was directed to find the proper handling procedure including treating and cycling which would permit fabrication of low-pressure panels 36 in. x 36 in. x 1/8 in. Proper regulation of oven drying temperature, flow and cure cycle will permit the making of large parts with no entrapment of volatiles.

Much work has been carried out on the treating of material on production size equipment and setting up of specifications. This made the material applicable for most production requirements on aircraft and guided missile construction where high strength at elevated temperatures is required. This work has also revealed certain relationships between various control factors necessary in treating and curing CTL-91-LD.

FACTORS AFFECTING PROPERTIES OF FINISHED LAMINATE

The glass is impregnated in a Waldron type treater in which the glass is passed into the resin pan, squeezed between nips and then is carried through the oven chamber where temperature is controlled by regulation of two zones. The material is allowed to cool before it is rolled. During this oven drying period the variables discussed below are controlled.

Inasmuch as phenolics react in condensation reaction with release of volatiles, the procedure of impregnation as well as fabrication is quite different from that of polyesters which are 100% solids resin with no release of volatiles in the polymerization process.

Flow

The flow tests were carried out with a flow tester which has a 1-1/2 in. diameter anvil electrically heated and controlled to maintain 350° F. A fixed pressure of 15 lbs. is applied to a press ram which meets the anvil and is also electrically heated and is controlled to a temperature of 350° F. The specimens used in the test are cut to one square inch. The specimens are inserted between sheets of cellophane and placed between the ram and the anvil. The pressure is applied for five minutes, then removed and the excess resin scraped off. The samples are previously weighed for original weight and they are then again weighed to determine the loss in resin.

The higher the flow the less tendency there is for moisture entrapment. As the resin is less viscous (at the plastic temperature) and begins to polymerize, the volatiles and products of condensation can escape. However, if the flow is too high, the mechanical properties at elevated temperatures are lower, possibly because of the fact that too much resin may be lost or because the resin does not fully polymerize to a hard mass. For higher pressure work, lower flows are used inasmuch as the higher pressure compensates for the lesser flow. This lower flow is gained by drying at a higher temperature or for a longer time or both.

Oven Drying Temperature

The temperature range in the Waldron Treater is very important in that it determines the flexibiltiy of the material after treatment. It has been found from experience that, in general, a material dried at 240° - 275° F. will suit most applications if the resin pick-up and flow are in specifications. These values are 40 - 45% resin pick-up and 27-30% flow. (see next section for details.) The explanation for varying degrees of stiffness lies in the difference in degree of polymerization and volatile content. The cycles by which panels can be made from materials impregnated using various treating conditions are described later.

The material dried at the higher temperature can be used in making direct compression large panels only by a low temperature cycle because of the resulting deterioration when this type of cycle is not used. The center is either discolored or roughened and lower elevated temperature properties are obtained in this instance. Inasmuch as this resin is further polymerized, upon heating in the press, the resin being more viscous, will not move freely as the lower temperature material. As condensation takes place, the moisture is trapped. Since the lower temperature, high-flow materials are less polymerized and less viscous, they flow more freely and flow for a longer time before the resin sets.

PROCESSING INFORMATION

The CTL-91-LD processed material is furnished on various grades or styles of glass fabric. The resin content and flow properties are controlled and various degrees of drape can be had. The flow of the material is determined by its end use. For low pressure work, higher flow material is available; for high-pressure work, lower flow materials are used. The material itself is in the form of a dry lay-up or B-stage laminating material. Various grades of glass may be impregnated e.g. 112, 116, 120, 128, 143, 181, 182, 184 (114 finish) with the resin.

The storage of CTL-91-LD processed material is very good under ordinary room temperature conditions. Only minor changes in the mechanical and electrical properties can be detected. Flexibility, however, is affected. At room temperature the material can be stored for several months and under refrigeration an indeterminate length of time. It is most desirable in order to maintain the drape characteristics of CTL-91-LD to store the impregnated fabric in a refrigerated room. Provision of a polyethylene inner-liner insures further maintenance of treated characteristics and protects the material against damage or sticking if the roll might be subjected to excess temperatures for a short period of time. Hanging the rolls on suitable racks will further insure the maintenance of treated specifications.

The various degrees of drape allow the laminations to be stretched or pulled over non-uniform shapes when warmed slightly by either an infra-red lamp or subjection to warming in an oven. The product enjoys the unusual facility of being molded at greatly varied pressures. Test work and experiences gained in the field indicate that pressures ranging from cellophane lagging through bag molding and positive pressures in the range of 10,000 psi are entirely satisfactory when the material is properly prepared for molding and properly postcured upon completion of the initial cure. Since the methods are so varied, each is listed below separately along with the most satisfactory curing cycles and postcuring cycles. It must be understood that the methods set forth are general methods and modifications may be necessary for specific or highly specialized applications.

Contoured Parts

Cellophane Lagging -

The use of cellophane lagging tape in the manufacture of heating ducts and other small parts is widespread. The method most often used is to prepare either break-away plaster forms or permanent coring of one type or another as the base upon which to build the part. The core should be coated with any standard releasing agent normally used in phenolic or polyester practice, preferably PVA. The CTL-91-LD is cut into narrow strips, the width depending upon the size and intricacy of the part to be prepared. These strips are then wrapped tightly around the form and the correct number of laminations provided to give the thickness required. When the lay-up is completed, cellophane tape is tightly wound around the CTL-91-LD. This assembly may then be placed in an oven and cured. To make a still more dense and more satisfactory part, the entire assembly may be placed in a rubber or PVA bag and subjected to vacuum in an oven or autoclave for curing. The cure used in this case is 275° F for 30 minutes. The part should then be removed from the oven, the bag and cellophane lagging removed and the core withdrawn. After cleanup, a postcure cycle at 250° F for 16-24 hours or a postcure utilizing progressive temperature increases and arriving at a final temperature of 400° -600° F may be used.

Bag Molding -

The bag molding method is similar to the one above for cellophane lagging with the exception of lending itself more readily to a large non-uniform contour than to the shapes often found in duct work. In the case of large parts with complex contours, the bag molding method is the most satisfactory. Either permanent molds or break-away plasters may be used. Releasing agents are required. In the larger and more complicated parts it is necessary to tailor sheets or laminations of CTL-91-LD to fit the shape of the part. In the areas where increased thickness is required or where reinforcing ribs are needed, more laminations or laminations of a thicker grade of glass are used. After the lay-up a perforated cellophane lamination or other suitable means of allowing gasses to escape should be fitted to the part. A lamination of glass fabric or cotton fabric is applied as a bleeder sheet as a further means of releasing gasses. Where extremely intricate contours are involved it is desirable to provide a fibrous mat on top of the glass fabric lamination to aid in distribution of vacuum pressures. The bag should then cover the entire assembly and the vacuum outlets be placed at strategic positions to affect the most satisfactory vacuum. The assembly is placed in an oven and cured in the same manner as suggested above.

Several modifications of this method have been used: One is to provide a second pressure bag outside of the vacuum bag. This provides additional pressure and a correspondingly stronger part. Another method commonly used is the evacuation within an autoclave so that higher pressures are attained. In this case, it is necessary to put heating coils within the autoclave (if the manufacturer wishes to use the PVA bag) as the moist steam and high temperature will destroy the bag and cause perforation and loss of vacuum. The pressure in the autoclave is controlled through use of externally compressed air. This is considered to be one of the most satisfactory methods of manufacturing large parts from CTL-91-LD.

Positive Pressure Laminating Using Matched Dies -

When there are great number of parts required, and the size is not prohibitive, it is desirable to mold in matched dies. In case of the compressor blades and other parts of the same approximate size, this is by far the best way to manufacture small parts. Dies may be made by any of the standard methods, such as electro-forming, pressure casting, straight tool machining, and so forth. The same general tooling practices as are used in the high pressure laminating or molding fields are desirable. It is suggested that dies or tools be either steam-heated or electrically heated and thermostatically controlled so that temperature may be maintained at the proper level.

In the case of small parts of approximately 4 to 10 square inches, rapid curing cycles can be evolved when temperatures are maintained at 400° F. A cure time of approximately eight minutes per one-eighth inch of thickness is satisfactory. The mold should be closed slowly so the heat may enter the plastic and exudation of excess resin and gaseous matter be attained. In this case the part may be withdrawn from the mold hot.

FABRICATION TECHNIQUES

Flat Panels -

The period of time necessary to reach the curing temperature of the resin has been found to be an important factor in obtaining panels with good appearance and high mechanical properties at elevated temperatures. This will have to be considered from two points of view; first, using a high flow material and the other, a low flow material. A low flow material can be made into a panel with good appearance if heated to cure temperature more rapidly than a corresponding higher flow material. Small parts such as compressor blades are made by a hot cycle and relatively low flow materials can be used. High flow material would tend to bleed too much and too great a loss of resin would result at the higher pressure (300 psi). In slower cycles an additional drying occurs as the laminate is being heated and low flow materials would become too viscous at the plastic temperature for proper movement which is necessary in order to obtain a good interlaminar bond.

Two general cycles may be used. One is called the standard cycle, the other the low temperature cycle.

Standard Cycle -

The laminate is inserted into a cold press and the pressure applied. The temperature is raised to at least 310° - 320° F and held for 15 - 30 minutes, depending on the thickness. The press is then cooled and the panel removed. In this cycle, the resin must remain sufficiently fluid for a long enough period to allow the volatiles to escape and the resin to distribute throughout the panel before the viscosity of the resin increases to the point where both volatile escape and resin movement are hindered. As indicated before, the initial stage of this cycle acts as a drying period to reduce flow, and aids in reducing residual volatiles which are those volatiles present in the form of solvents, free phenol, etc. This period can be varied according to the pressure and the material being used. The initial period can also be hastened by preheating the press before the laminate is inserted.

After this period, the resin becomes liquid and as the temperature increases, further polymerization and cure take place. It is desirable to keep the resin in this liquid stage as long as possible in order to permit better distribution of the resin and release of volatiles. This can be achieved by using higher flow materials, a shorter drying period in the press, and higher pressures. It is desirable to release the volatiles caused by polymerization slowly over a long period before the resin sets and entraps products of condensation.

If top cure temperature in the press does not reach 310° - 320° F, a postcure is necessitated. For example, the following postcure may be used, namely, 24 hours at 300° F; 12 hours at 350° F; and 1/2 hour each at 400° F and 500° F.

Low Temperature Cycle -

In this cycle, the low flow material is put into a cold press and pressures up to 30 psi are applied. The press is heated to a temperature (usually 250° F) approximately 10° - 15° F cooler than the highest temperature at which the material was treated. This temperature is held for one-half hour and the laminate cooled; the laminate is then postcured for 24 hours at 250° F, 12 hours at 300° F, and 12 hours at 350° F, or some similar postcure. The larger the area or the thicker the section, the slower the postcure.

This cycle reduces the amount of volatiles released in the press since the material was initially treated at a higher temperature than it is subjected to in the press. The postcure cycle serves to further the polymerization and allows the slow release of volatiles. This cycle could be applied to any size part and only postcure time need be varied.

USES OF CTL-91-LD

Material can be successfully molded at high pressures (250 psi) and maintain as high mechanical properties at elevated temperatures in very small sizes as those panels pressed at 15 psi.

On compressor rotor blades submitted to Wright Field the following data were obtained on a molded blade using alternating lamination of 181-114 and 143-114 glass fabric with 113-114 surfaces:

Flexural	Room Temperature	1/2 Hr. at 500° F
Ultimate Strength psi	60,400	44,400
Modulus of Elasticity, psi x 10^6	4.37	3.07

For this application, tests disclosed that CTL-91-LD laminates exhibited extremely good fatigue resistance and damping superior to the metal counterpart.

In order to check the effect on flexural tests of the angle at which the laminations lie in the blade, specimens were cut from 1 inch x 24 inch strips of flat panels which had been pressed at 150 psi. Tests were made at room temperature. In column I below are the test results with the testing pressure applied perpendicular to the laminations, and in column II are the test results when the specimens were cut from the strips on a taper so that they were made more nearly comparable to those specimens cut entirely from the finished blade.

	I	II
Flexural Ultimate strength psi	86,000	66,900
Flexural Modulus of Elasticity psi x 10^6	4.78	6.48

In addition to the above, the CTL-91-LD resin has been used successfully in making of honeycomb. Compression tests at room temperature on various types of honeycomb, manufactured by Western Products, Inc. have been conducted by Forest Products Laboratory. Preliminary data obtained are listed below.

Results of Tests made at Forest Products Laboratory on Honeycomb Core Material Manufactured by Western Products, Inc. using CTL-91-LD

Density	Cell Size	Average Compressive Strength (psi)	Average Modulus of Elasticity (psi)
5 lbs/cu. ft.	3/16 in.	618*	126,300
5 lbs/cu. ft.	3/16 in.	637**	129,800

*Compression tests were made on specimens 2 inch x 2 inch x 6 inch with the ends set 3/8 inch into plaster and conditioned to equilibrium moisture content in a room maintained at 74° F and 65% humidity. Strains were measured over a two inch gage length using a Marten's mirror apparatus.

**Specimens conditioned to equilibrium moisture content in a room maintained at 80° F and 97% humidity.

The high strength at elevated temperatures along with the low specific gravity of the material invited investigation of many other applications.

These included parts for the guided missile and aircraft manufacturers. In each case the CTL-91-LD laminate appeared to satisfy the requirements. Guided missile manufacturers have used the material in an experimental electronic radome in the guided missile control system and have fabricated many parts including structural sections such as fins, wings, bulkheads, telemetering windows, etc. The aircraft groups have made air scoops, wing tips, radomes, water tanks and various heating ducts for use in de-icing systems. (see WADC Data Figure i)

TEST DATA

Test data are summarized in Table I showing the various size panels, cycles, and resulting flexural strengths. Long-time temperature tests (Appendix VI) are included as well as some preliminary data on various finishes (Appendix V.)

In addition to the data listed in Table I, some typical data are listed in Table II. Table III indicates some of the electrical properties.

PHYSICAL DATA OBTAINED FROM CUSTOMER RESEARCH

The test programs under way in other laboratories have been reported to us in many cases. It is apparent that the data set forth in Tables II and III is typical only when general curing and postcuring methods are used. In most cases it is possible to improve the properties of the material when studied for a specific part or application. This fact is understandable when it is considered that the customer laboratories can devote more time to the perfection of a single part than our engineering department. For instance, one missile manufacturer has reported values of the following order -

Flexural Strength at room temperature, 65,000 - 75,000 psi;

Flexural Strength at 550° F after 1/2 hour exposure at 550° F, 47,000 - 57,000 psi;

Modulus of Elasticity at room temperature, 4.1 million;

Modulus of Elasticity at 500° F, 3.5 million.

A flexural strength of more than 19,000 psi is indicated after eleven minutes at 850° F, and 30,000 psi after three and one-half minutes at 750° F. This information does not suggest that the material be used at these temperatures for long periods of time; it does indicate that it is satisfactory for the flight-time of most of the faster missiles now in production or under development. (See Appendix III.)

In the guided missile field we have also had reports that rocket motors and blast tubes have been successfully tested. The blast tubes have been subjected to 2000° F for as long as 80 seconds with only minor erosion taking place. (See Appendix III)

We have also had reports from aircraft laboratories indicating that CTL-91-LD has higher long-time heat resistance than the values taken from our own laboratory reports. Flexural strengths of 40,500 psi after 200 hours at 450° F and flexural strength of 33,000 psi after 200 hours at 500° have been obtained. (See Appendix VI)

CURRENT DEVELOPMENT PROGRAM

The current development program is aimed at improving the heat resistance of laminates made with CTL-91-LD for both short-time and long-time applications. This is being attempted by the use of additives to the resin and varying processing, curing and postcuring techniques.

The indications at the present time are that the panels giving the highest short time flexural values at elevated temperatures are not necessarily those that result in the highest values when subjected to temperatures for longer periods.

Tests are to be conducted to determine whether there is a critical temperature where the mechanical properties drop at a more rapid rate.

Another objective is aimed at determining if there is a relationship that will indicate what might be expected for long-time tests at elevated temperatures. An attempt will be made to determine whether the relationship is between resin remaining, or resin remaining and density, or resin density, and long-time flexural strength at elevated temperatures. For short-time tests, the indications are that good results can be obtained with a variety of resin remaining values, some as low as 15%.

Results on the above tests will be reported at a later date.

APPENDIX I

COMPRESSION DATA ON CTL-91-LD LAMINATES

Some specialized compression tests have been performed and the following is a description of one such specialized test, performed by a user.

The test was performed on a tube. The OD of the tube was 6.40 inches. The wall thickness was .040 inch. The tube was made by expanding a convolutely wound preform against a closed female mold, which carried the correct OD dimension. Expansion was secured by inflating a rubber bag inside the preform, much as the innertube of a tire is expanded against the casing. The manufactured length of tubing was 37 inches. The actual length of the section tested was 18 inches. The test conditions were as follows:

> An 18 inch length of tubing, which had been carefully cut to secure edges which were relatively undamaged and whose plane was perpendicular to the axis of the tubing, was placed in a testing machine. The compression load was applied directly parallel to the axis of the tube. Head travel was at the rate of .050 inch per minute. Failure occured at a total compression load of 13,800 pounds. Young's Modulus in compression was determined to be 4.6 x 10^6. Failure was a combination of buckling and shear, occuring along lines in the outside surface of the tube, the angle of principal action being at 45°.

This tube was tested at room temperature. Actual molding conditions were 300 psi and 325° F. The material was 91-LD on 181-114 finish glass fabric.

APPENDIX II

THERMAL DATA *

1. Thermal conductivity, k = 0.15 BTU/hr./ft/degrees F.

2. Thermal diffusivity, = 5.6 x 10-3 BTU/hr/ft.^{2}

3. Specific heat, C = 0.23 BTU/lb./degrees F.

4. Density = 115 lb./ft.^{3}

5. Coefficient of thermal expansion = 3.2 x 10-6 /degrees F. for 181-114 cloth pressed @ 15 psi.

This coefficient is for temperatures ranging from 70° F to 450° F. If the cloth (181) is interleafed with 143 cloth the coefficient becomes 3.59 x 10-6 degrees F. in a direction parallel to the 143 cloth.

The thermal diffusivity coefficient was determined at a mean temperature of 350 - 400° F on a specimen 0.125 inch thick. Lower values were obtained for a thinner specimen but it is believed the 0.125 sheets yielded results that are more accurate.

6. Molding temperature, pressure and curing temperature:

The panels are baked between 275° F and 325° F at 50-250 psi for 30 minutes. They are then cured for 16 hours at 275° F, followed by an increase in temperature to 400° F at 50° F per hour. The specimen is then held at 400° F for one hour.

The data given in item (6) is for panels 0.060 in. x 8 in. x 12 in. Temperature, time, and postcure process depend to a large extent on the dimensions of the piece being molded. The postcure process yields higher physicals at elevated temperatures, which is accompanied by a loss in ductility.

* Hughes Aircraft Data

APPENDIX III

Spot Check Tests at Temperatures Above 500° F.

Check #1 -

This covers static pressure tests at only 500° F.

Part:	At chamber, closed at one end, open and threaded at the other end.
Wall Thickness:	.110 inches
Diameter:	5 inches
Length	20 inches
Static Pressure Test:	1,000 psi at 500° F
Short Time:	20 minutes
Results:	Part passed test successfully.

Check #2 -

Restriction flow parts

Part:	Small cylindrical prism with hole through the center, cross section of hole circular but varies in diameter.
Conditions of firing Test:	High velocity gas stream at 3,700 degrees F.
Time:	4 seconds.
Results:	No visible erosion on surface exposed to high velocity flow. Physical properties after firing estimated to be 75% of original room temperature properties.

Check #3 -

Part:	Same as used in Check #2
Conditions of Firing Test:	High velocity gas stream at 2,000 degrees F.

Time:	60 seconds.
Results:	No visible erosion on surface exposed to high velocity flow. Physical properties estimated to be 85% of original room temperature properties.

Check #4 -

Gasket Applications

Conditions of Firing Test:	Edge of gasket exposed to high velocity hot gas stream.
Firing Conditions:	Temperature of gas stream 3,250 degree F.
Time:	2-1/2 seconds
Results:	Essentially same as Check #2. No visible erosion or sign of delamination.

Check #5 -

Flame Barrier

Part:	1/4 inch thick laminated sheet.
Conditions of Test:	One side of sheet exposed to hot gas @ 2,000° F. Other side exposed to room temperature.
Time:	60 seconds
Results:	Hot surface of panel carbonized. Panel still physically intact. No blisters or delamination.

Note: Check Tests 1 - 5 performed on parts made by Procurement Laboratory.

Additional information covering behavior at temperatures above 500° F: To date, no systematic evaluation of properties at temperatures above 500° F. have been carried out. Some spot checks have been made of:

Flexural strength @ 750° F. (after exposure to 750° F for 11 minutes) 19,000 psi.

Note: Specimen was cut from panel prepared by Cincinnati Testing and Research Laboratories. Above test was carried out at Lockheed Aircraft Corporation.

Check #6 -

Part: Standard LP 406, Flexural Specimen.

Conditions of Test: Flexural tested @ 600° F after exposure to 600° F for 1/2 hour.

Results: Ultimate flexural strength 32,000 psi. Surface of test specimen carbonized. No apparent physical deterioration of sheet visible due to temperature, other than carbonization at surfaces.

Note: Check #6 performed by Structures Research Section, Aerophysics Division, North American Aviation, Inc.

Some qualitative tests have been performed by Consolidated Aircraft on the effect of high velocity airstreams at elevated temperatures.

A reinforcing pad made by Hughes Aircraft Company was tested by Consolidated. The pad was placed in an airstream of velocity Moch 2.2, temperature of airstream 750° F, for a total of 90 seconds. The part was placed end-wise in the airstream under the most unfavorable conditions, (i.e. principal stress tending to delaminate.) The raw edge of the molded part displayed no sign of mechanical deterioration.

APPENDIX IV

A PRELIMINARY EVALUATION OF HIGH HEAT RESINS

The following report presents information on compressive strength at elevated temperatures of honeycomb material dipped in various resins and was furnished by California Reinforced Plastics Company.

TEST REPORT #601

1. This report covers tests conducted to determine the strengths at elevated temperatures of various resins when used as dip resins for glass fabric honeycomb. Compression tests on honeycomb panels were conducted at temperatures up to 600° F.

2. Test Methods

   California Reinforced Plastics Hexcel glass fabric honeycomb is fabricated at low density, and is normally dipped in polyester resin to increase strength by increasing the density of the material. In these tests the regular low density NP core was dipped to weight in the resin to be evaluated; therefore, the base glass fabric was the same, the only difference was the dip resin.

   Heated platens were mounted in the testing machine and specimens were placed between the platens at contact pressure for twenty minutes prior to testing. This allowed the specimens to come to test temperature. The specimens were then tested to failure in flat compression. Thermocouples were inserted in preliminary specimens to insure that the specimens reached temperature in the twenty minutes allowed.

3. Specimen Preparation

   Tests were conducted in both bare compression and panel compression. The bare specimens were sawed to 0.500 inch thickness, cut to 2 inch x 2 inch square, and tested as above. The panels were prepared as follows:

   (a) Core Preparation: Cut to 0.500 inch thickness, edge dipped in CTL resin and air dried for 24 hours. Oven dried for four hours at 180° F.

   (b) Skin Preparation: Two ply of 181 glass cloth per face were dipped in CTL resin and cured to advance "B" stage.

   (c) Panel: Laid up and cured at 20 psi and 350° F. for one hour.

   Test coupons were cut 2 in. x 2 in. square and tested as above.

4. Conclusions:

   Four different dip resins were evaluated as well as a panel of

NP-1/4-21-8.0 with a polyester dip which was used as a control. A total of 169 bare specimens and 142 panel specimens were tested. The significant results are tabulated below, and plotted in Figure 2. Note that some specimens were conditioned for one hour at 600° F before crushing, and these specimens had a higher crushing strength than those conditioned twenty minutes.

The only resin tested which showed considerable retention of strength at elevated temperature is that designated at CTL. The remainder approached the strength of the control panel as temperature was increased.

Crushing strength of 0.500 in. Thick Panels in Pounds per Square Inch.

Type	Temperature - Conditioned 20 minutes				Conditioned 60 Minutes
	70° F.	300° F.	500° F.	600° F.	600° F.
CTL 1/4-21-8.0	1765	1008	656	605	657
SBL 1/4-21-8.0	1178	750	313	107	128
MS 1/4-21-8.0	1479	380	160	109	156
16B 1/4-21-9.0	985	293	207	119	151
NP 1/4-21-8.0	1240	234		85	

On the basis of these tests California Reinforced Plastics Company will produce a new type Hexcel designated CTL in 3/8, 1/4, and 3/16 cell sizes, in density ranging from 4.0 to 9.0 pounds per cubic foot. This heat resistant honeycomb will be produced on special order.

This company will continue to evaluate high temperature resins, with particular attention to length of exposure at elevated temperature, and results of further tests will be made available to the industry.

APPENDIX V

PRELIMINARY EVALUATION OF THE EFFECT OF VARIOUS FINISHES ON GLASS CLOTH ON LAMINATES USING CTL-91-LD

The following preliminary data on the various finishes on glass cloth were obtained on bars using 181 fabric and CTL-91-LD resin. The bars were cured at 350° F for 20 minutes at approximately 300 psi pressure. The material was placed in the hot mold, cured, and discharged hot. The bars were approximately 3/4 in. wide and 24 in. long. Some tests were also made on specimens taken from 8 in x 12 in. x 18 in. panels pressed in the standard cycle described in an earlier part of this report.

These results are indicative only. Some of the data represents only one or two tests; others represent several tests on bars with varying numbers of laminations.

As indicated on the enclosed table, the caramelized (pyrolized glass-no finish) yielded high room temperature flexural values and good flexurals after 1/2 hour at 500° F tested at 500° F but was extremely poor after the two hour boil. The 114 finish had better values after the two hour boil than the Garan and in most cases the 136. The Volan finish appears to be the best in the two hour boil, even better than the Bjorksten.

As far as the one-half hour heat tests are concerned, there is much overlapping. This is due to variations in numbers of laminations but the range does indicate that the 114 holds up well although the Garan, 136, and Linde show some higher values.

The Linde GS-1 finish indicates excellent heat resistance with fair water resistance, and the Volan excellent water resistance, with good heat resistance. Bjorksten finish shows good water resistance. In general, the 114 and Volan hold up well under all the conditions.

FLEXURAL [illegible]
(psi)

HAND TREATED BARS

Type	R. T.	1/2 HR. 500° F	2 HOUR BOIL
Garan	66,000 - 66,500	45,100 - 46,800	32,300 - 39,350
Bjorksten	53,900 - 56,500	46,050 - 46,500	53,000 - 50,350
136	61,000 - 72,500	46,350 - 56,500	39,150 - 47,750
114	58,000	48,850	47,500
Volan	65,900 - 74,000	46,100 - 53,900	58,400 - 65,900
Linde (GS-1)	61,900 - 62,900	54,800 - 60,850	43,100 - 45,250

MACHINE TREATED - BARS

Type	R. T.	1/2 HR. 500° F	2 HOUR BOIL
Garan	52,000 - 66,500	46,600 - 60,950	36,150 - 42,250
Caramelized	61,000 - 78,500	49,250 - 67,000	7,000 - 12,200
114	57,500 - 69,250	47,300 - 60,100	48,950 - 61,650

MACHINE TREATED-PRESSED STANDARD CYCLE

8 in. x 12 in. x 1/8 in.

Garan	49,500	53,800	36,700
Caramelized	58,500	48,450	20,200
114	53,000	44,450	46,050

APPENDIX VI

LONG-TIME ELEVATED TEMPERATURE FLEXURAL TESTS

Flexural tests were carried out at 400° F and 500° F after exposures up to 200 hours at temperature. The results are shown on Figure 5.

The panels (9 in. x 13 in. x 1/8 in.) were made from 12 laminations of CTL-91-LD having a 40% resin content and 30% flow. The build-up was wrapped in cellophane and inserted into the press which had been preheated to 265° F. The press was closed to contact pressure and held for 1-1/2 minutes. The pressure was then raised to 160 psi and held for 8 minutes. The panel was then removed from the press, the cellophane stripped off and the postcuring cycle begun.

The postcure was 24 hours each at 250° F, 300° F, and 350° F. Upon completion of the postcure, whole panels were placed in the exposure ovens; one panel for each exposure period. At the end of each exposure period, the panel was removed, trimmed and test specimens prepared.

Each point on the graph represents an average of 8 test specimens.

It should be noted that these values are much higher than those shown on Figure 4 which represents values obtained on 15 psi panels cured in the standard cycle.

The improved values obtained on panels pressed by the cycle described above are due in part to the lack of porosity which deters oxidation as compared to the lower pressure panels. A second point is that the low pressure panel specimens were exposed as specimens (and not as whole panels) which permitted more rapid oxidation.

The room temperature flexural values and 1/2 hour exposure flexural values on the standard cycle panels are better, but the long-time exposure flexural tests are not as good as the higher pressure panels.

Hence, where the application is for long-time exposures at elevated temperatures the above cycle is better.

APPENDIX VII

WRIGHT AIR DEVELOPMENT CENTER
EVALUATION PROGRAM

The following tests are to be carried out under the WADC evaluation program for CTL-91-LD.

1. Flexural versus temperature and time of exposure. (National Bureau of Standards)

2. Tension and compression versus temperature after one-half hour and 200 hours at temperature. (Batelle Memorial Institute)

3. Tension creep rupture and compression creep rupture (one to one thousand hours versus temperature) (Batelle Memorial Institute)

4. Fatigue and damping capacity tests versus temperature are planned. (University of Minnesota)

TABLE I

SUMMARY OF PROCESSING, CYCLING AND FLEXURAL TEST RESULTS ON VARIOUS SIZE PANELS

	MAX. AREA	MAX. THICKNESS INCHES	FLOW & TEMPERATURE RANGE TREATING (PERCENT)	RESIN CONTENT RANGE TREATING (PERCENT)	PRESSURE	CURECYCLE	POSTCURE CYCLE	FLEXURAL AT 500°F AFTER 1/2 HOUR @ 500°F (PSI) EDGE	CENTER	FLEXURAL AT ROOM TEMPERATURE (PSI) EDGE	CENTER	RESIN REMAINING (PERCENT)	SPECIFIC GRAVITY
JET ENGINE BLADES ALTERNATE 181-114 & 143-114	1-1/2" x 6"	.125	20-23 at 220°F or 28-30 at 260°F (181-114) 14-16 at 220°F (143-114)	38-39 at 220°F or 45-48 at 260°F 181-114) 35-38 at 220°F (143-114)	300 PSI CLOSED MOLD	8 MINUTES @ 350°F INSERTED HOT	NONE	H.49,000 L.39,000 A.44,000		H.79,000 L.69,000 A.74,000		23.5	1.70
TEST BARS ALL 181-114	1" x 24"	.250	20-23 at 220°F or 28-30 at 260°F	38-39 at 220°F or 45-48 at 260°F	300 PSI CLOSED MOLD	20-25 MINUTES @ 350°F INSERTED HOT	NONE	A.46,300[a]		A.63,200[a]		25.8	1.76
TEST BARS ALL 143-114	1" x 24"	.250	14-16 at 220°F	35-38 at 220°F	300 PSI CLOSED MOLD	20-25 MINUTES @ 350°F INSERTED HOT	NONE	A.56,400[a]		A.91,400[a]		22.8	1.78
TEST BARS ALTERNATE 181-114 & 143-114	1" x 24"	.260	COMBINATION OF THE TWO ABOVE	COMBINATION OF THE TWO ABOVE	300 PSI CLOSED MOLD	20-25 MINUTES @ 350°F INSERTED HOT	NONE	A.56,500[a]		A.85,300[a]		25.4	1.73
	8" x 12"	.065	30-33 at 260°F	48-50 at 260°F	125 PSI OPEN PRESS OR WITH STOPS TO SIMILATE CLOSED MOLD	7 MINUTES @ 260°F INSERTED HOT	12 HRS.AT 260°F 8 HRS.AT 300°F 1/2 HR.EACH @ 350°F, 400, 450° and 500°F	H.44,300 L.28,800 A.40,775		H.61,400 L.49,200 A.56,800			
	12" x 12"	.125	20-30 at 220°F	38-39 at 220°F	15 PSI OPEN PRESS	15 MINUTES @ 350°F INSERTED COLD, BACKED BY STEEL PLATES & CARDBOARD	NONE	H.44,600 L.39,700 A.41,900	H.41,400 L.38,800 A.40,200	H.60,900 L.54,100 A.59,100	H.57,700 L.51,500 A.54,400	27.5	1.58
	12" x 12"	.125	28-30 at 260°F	45-48 at 260°F	35-40 PSI OPEN PRESS	30 MINUTES @ 260°F INSERTED COLD, BACKED BY STEEL PLATES	12 HRS.AT 250°F 8 HRS.AT 300°F 4 HRS.AT 350°F	H.42,400 L.39,900 A.40,800	H.43,000 L.39,000 A.41,200	H.59,600 L.52,900 A.56,800	H.60,200 L.52,600 A.57,400		
	18" x 18"	.125	23-26 at 200°F	40-41 at 220°F	15 PSI OPEN PRESS	15 MINUTES @ 350°F INSERTED COLD, BACKED BY STEEL PLATES & CARDBOARD	NONE	H.44,000 L.40,200 A.42,000	H.40,100 L.36,200 A.37,800	H.64,300 L.60,400 A.59,200	H.62,100 L.59,300 A.56,000	27.8	1.67
	18" x 18"	.125	30-33 at 260°F	48-50 at 260°F	35-40 PSI OPEN PRESS	30 MINUTES @ 260°F INSERTED COLD, BACKED BY STEEL PLATES.	12 HRS.AT 250°F 8 HRS.AT 300°F 4 HRS.AT 350°F	H.42,600 L.38,500 A.40,400	H.43,000 L.37,000 A.40,250	H.62,100 L.53,300 A.58,000	H.63,200 L.53,000 A.58,400		
	12" x 12"	.125	20-30 at 220°F	38-39 at 220°F	1100 PSI	15 MINUTES @ 350°F INSERTED COLD, BACKED BY STEEL PLATES	NONE	H.33,000 L.23,000 A.27,400	H.30,700 L.25,500 A.27,900			30.1	1.58
	20" x 21"	.050	23-26 at 220°F	40-41 at 220°F	18-20 PSI OPEN PRESS	15 MINUTES @ 350°F INSERTED HOT, BACKED BY STEEL PLATES	NONE	H.43,900 L.40,400 A.42,560	H.36,200 L.34,600 A.35,000				
WADC DATA SEE NOTE b	10" x 10"	.112	20 at 240°F	38-42 at 240°F	30 PSI	25 MINUTES @ 300°F	NONE	43,750		47,250			
WADC DATA	36" x 36"	.144	35 at 260°F	45-49 at 260°F	BAG MOLDED[c] SEE NOTE	1 HR.20 MINUTES @ 309°F	NONE 22 HRS.265-275°F 45 MIN.325°F 15 MIN.450-500°F 3 HRS.250-300°F 2 HRS.300-350°F 15 MIN.400°F 15 MIN.450-500°F	43,060[d] 36,500[e] 47,350[d] 36,130[e] 45,200[d] 36,970[e]		54,640[d] 50,100[e]			
NORTH AMERICAN AVIATION DATA	36" x 36"	.144	35 at 260°F	45-49 at 260°F	BAG MOLDED[c] SEE NOTE	1 HR.20 MINUTES @ 309°F	17 HRS. 275°F 2 HRS. 400°F 2 MIN. 510°F 1/2 HR. 275°F 17 HRS. 275°F 2 HRS. 400°F	H.50,300 L.47,200 A.49,170 41,900 38,600	H.2.20[f] L.1.91 A.2.06 1.775 1.77			34.5	1.65

NOTES:

H = HIGH

L = LOW

A = AVERAGE

a - THESE FIGURES ARE THE RESULTS OF TESTS MADE BY THE MATERIALS LAB., AND ONLY AVERAGE FIGURES WERE MADE AVAILABLE.

b - COMPRESSIVE 31, 600 PSI.

c - PANEL WAS BAG MOLDED BY NORTH AMERICAN AVIATION, INC. USING 12 LAMINATIONS LAID UP ON AN ALUMINUM PLATE WITH COARSE GLASS CLOTH AND FELT USED AS A BLEEDER, PLUS A RELEASING AGENT. THE PACK WAS BAGGED IN PVA WITH ZINC CHROMATE PUTTY USED FOR SEALING THE EDGES.

d - SPAN - PARALLEL TO WARP.

e - SPAN - PERPENDICULAR TO WARP.

f - ELASTIC MODULUS IN FLEXURE

TABLE II

TYPICAL MECHANICAL PROPERTIES OF CTL-91-LD OBTAINED
ON PANELS PRESSED AT 15 PSI

PROPERTY	77° F	Tested at 500° F after 1/2 Hr. at 500° F
Flexural Strength - Ultimate	55,000 psi	40,000 psi
Flexural Modulus of Elasticity	3.0×10^6 psi	2.5×10^6 psi
Flexural Strength after 24 hr. immersion water	40,000 psi	
Flexural Modulus of Elasticity, 24 hrs. water	2.75×10^6 psi	
Tensile Strength, Ultimate	40,000 psi	35,000 psi
Tensile Modulus of Elasticity	2.5×10^6 psi	
Compressive Strength - Edgewise	40,000 psi	
Impact Strength (Izod ft-lb/in. notch)	13.5	
Specific Gravity	1.5 - 1.8 *	
Resin Content - cured, laminated	25 - 35% **	

* Molded at 15 psi, bag or open mold, 181-114 glass fibre, panel size 36 in. x 36 in.

** Depending upon mold pressure and flow.

TABLE III

ELECTRICAL PROPERTIES

One Megacycle

	Room Temperatures	24 Hrs. in water
Power Factor	0.0137	0.0171
Dielectric Constant	4.39	4.39
Loss Factor	0.0601	0.0752

These properties will vary with the cycle, the material, etc. Power factor values in the range of .01 - .014 can be obtained at 1 mc. The dielectric constant will range from 4.00 - 4.4 at 1 mc. The above values were obtained at 1 mc, on specimens taken from an 18 in. x 18 in. x 18 in. panel made at 15 psi in the standard cycle.

WADC DATA FIGURE 1

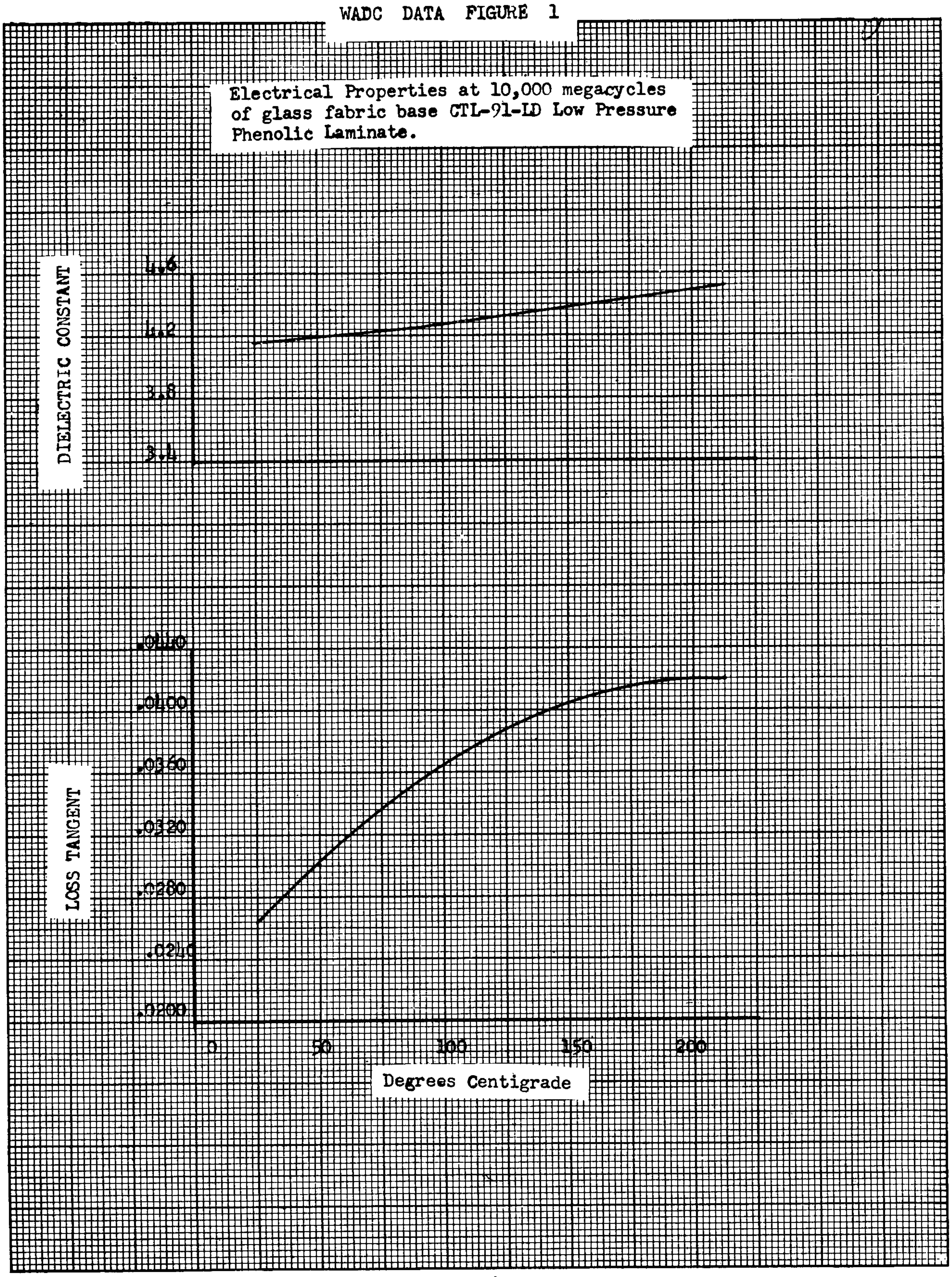


FIGURE 2

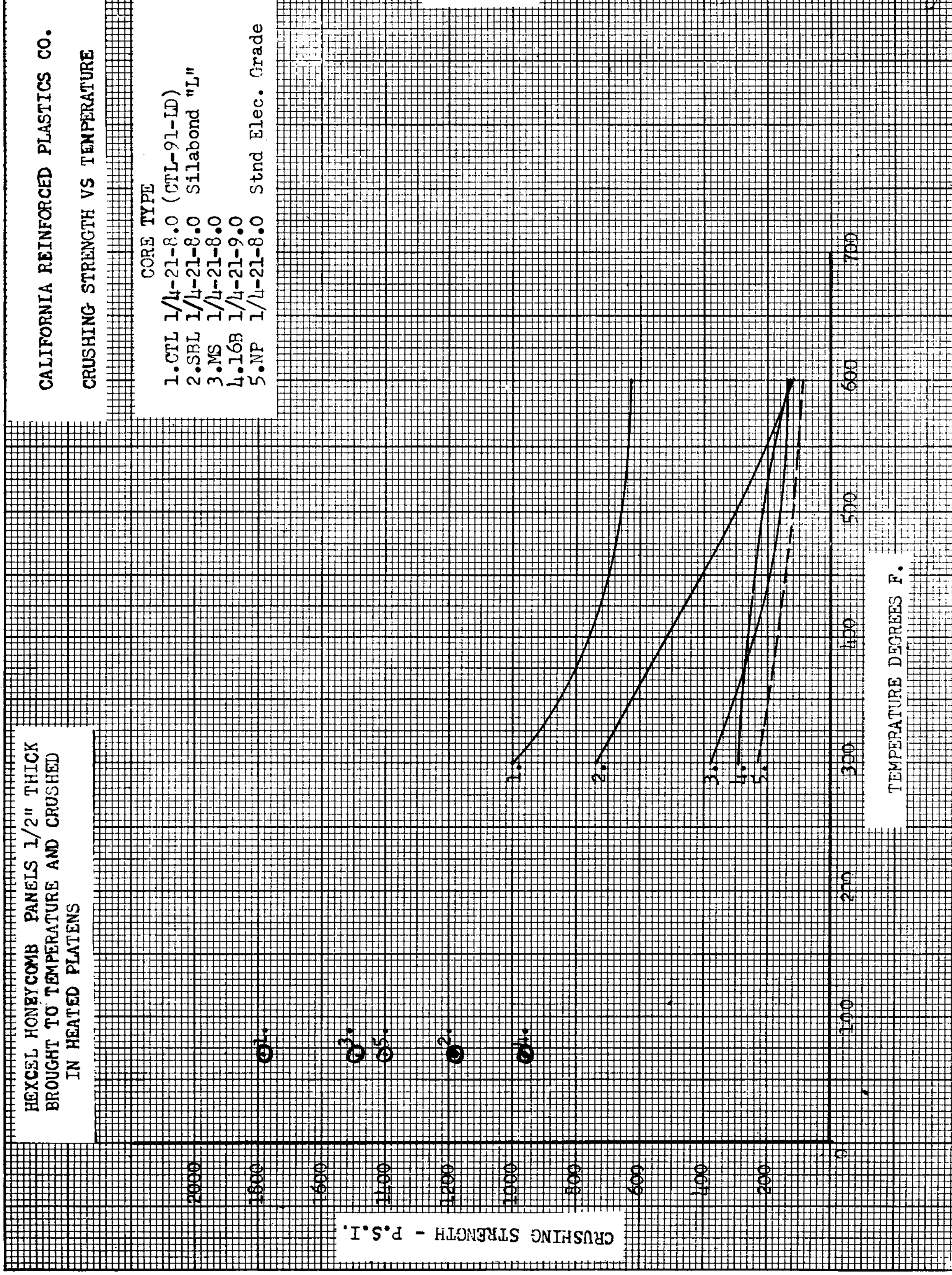

CALIFORNIA REINFORCED PLASTICS CO.
CRUSHING STRENGTH VS TEMPERATURE
CORE TYPE
1.CTL 1/4-21-8.0 (CTL-91-LD)
2.SBL 1/4-21-8.0 Silabond "L"
3.MS 1/4-21-8.0
4.16B 1/4-21-9.0
5.NP 1/4-21-8.0 Stnd Elec. Grade
HEXCEL HONEYCOMB PANELS 1/2" THICK
BROUGHT TO TEMPERATURE AND CRUSHED
IN HEATED PLATENS
CRUSHING STRENGTH - P.S.I.
2000
1800
1600
1400
1200
1000
800
600
400
200
TEMPERATURE DEGREES F.
0
100
200
300
400
500
600
700

FIGURE 3

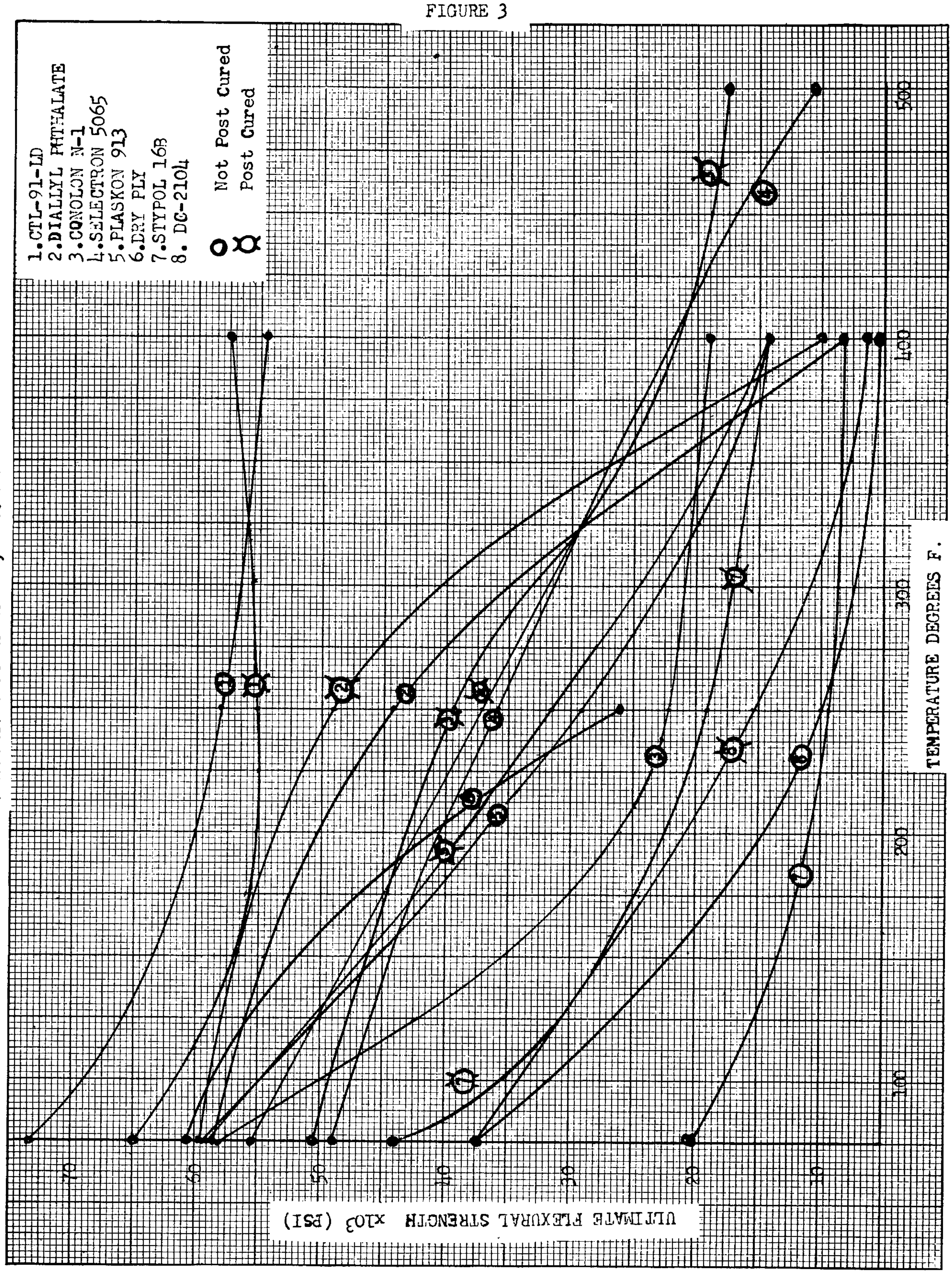

1.CTL-91-LD
2.DIALLYL PHTHALATE
3.CONOLON N-1
4.SELECTRON 5065
5.PLASKON 913
6.DRY PLY
7.STYPOL 16B
8. DC-2104
Not Post Cured
Post Cured
NORTH AMERICAN AVIATION, INC. DATA
TEMPERATURE DEGREES F.
100
200
300
400
500
ULTIMATE FLEXURAL STRENGTH x10^3 (PSI)
70
60
50
40
30
20
10

FIGURE 4

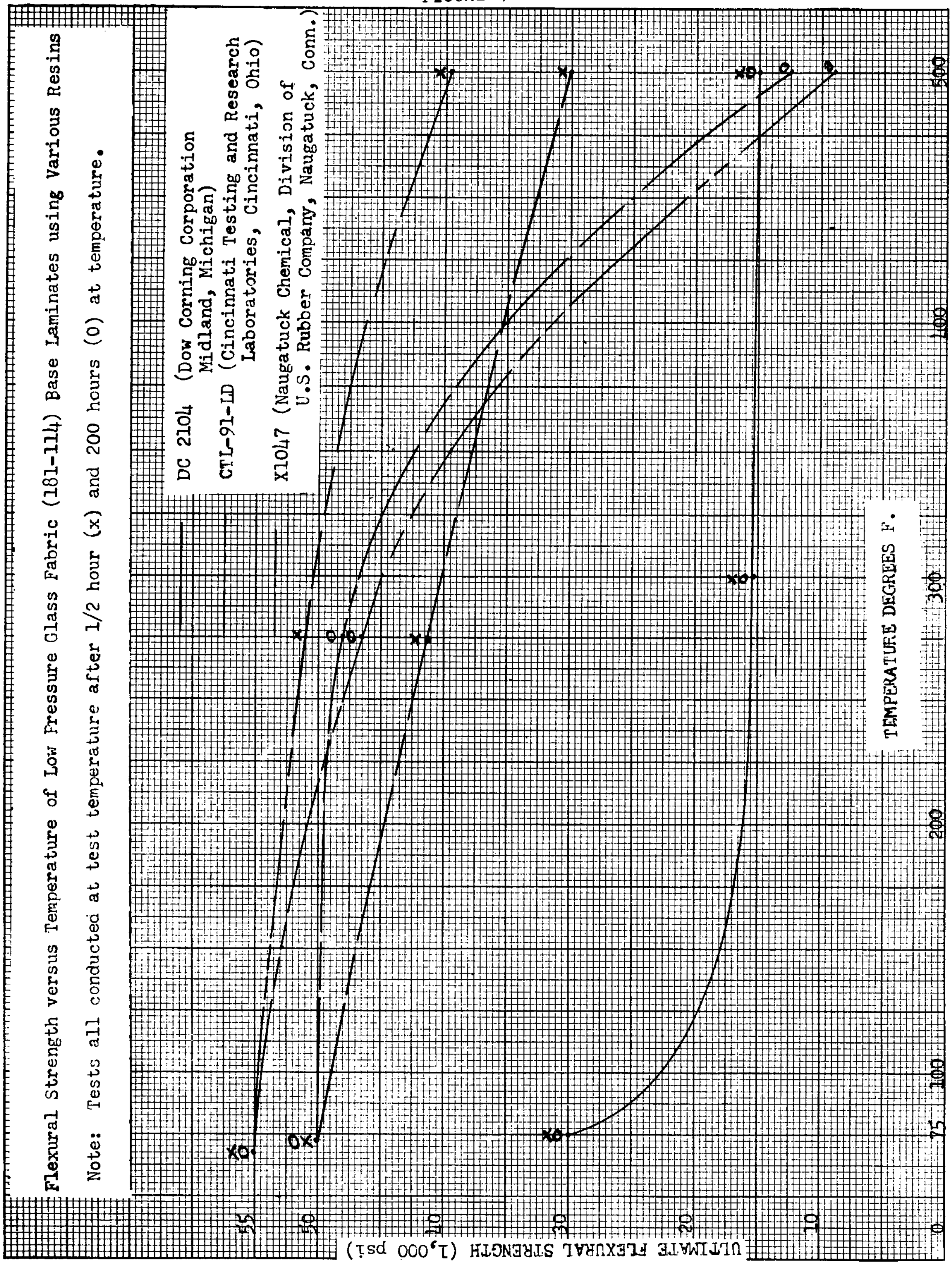
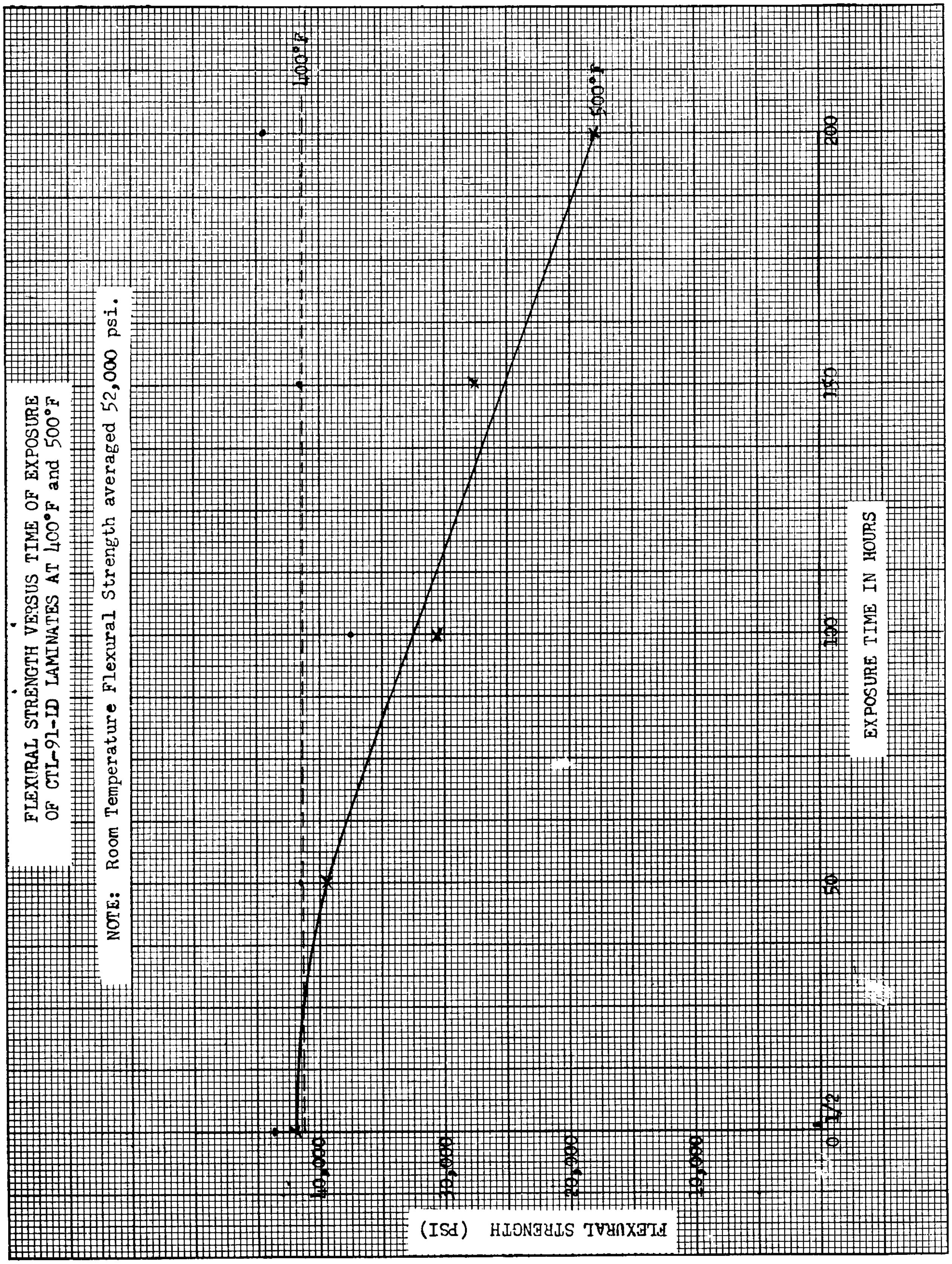

Flexural Strength versus Temperature of Low Pressure Glass Fabric (181-114) Base Laminates using Various Resins
Note: Tests all conducted at test temperature after 1/2 hour (x) and 200 hours (0) at temperature.
DC 2104 (Dow Corning Corporation Midland, Michigan)
CTL-91-LD (Cincinnati Testing and Research Laboratories, Cincinnati, Ohio)
X1047 (Naugatuck Chemical, Division of U.S. Rubber Company, Naugatuck, Conn.)
TEMPERATURE DEGREES F.
0
75
100
200
300
400
500
ULTIMATE FLEXURAL STRENGTH (1,000 psi)
55
50
40
30
20
10

FIGURE 5


FLEXURAL STRENGTH VERSUS TIME OF EXPOSURE
OF CTL-91-LD LAMINATES AT 400°F and 500°F
NOTE: Room Temperature Flexural Strength averaged 52,000 psi.
400°F
500°F
EXPOSURE TIME IN HOURS
0
1/2
50
100
150
200
40,000
30,000
20,000
10,000
FLEXURAL STRENGTH (PSI)